

**Audit of Data Quality
March 2009 Sampling Event
Headspace Methane Data
Data associated with “Ground-Water Investigation in Pavillion, Wyoming,” QA ID #G-14478
analyzed at US EPA Region VIII Laboratory
ADQ performed by Neptune and Company, Inc.**

ADQ Report Date: January 19, 2011 – Revision 1.

This revised report completes the audit of the March 2009 sampling event with the Headspace Methane analysis. The first report provided included completed audits of data quality for Semivolatiles, Alkalinity, Anions, and DRO. The first audit of data quality report was incomplete with respect to the headspace methane data. To complete that audit, additional information was requested from EPA Region 8. The information has been received from EPA Region 8 and was used to complete this report. For this report, a validation Excel spreadsheet for TO61 WO5 Headspace Methane Validation Worksheets is included. This worksheet includes documentation of the validation process, along with sample and batch information, and recalculations. This report completes the audit of data quality for all samples and parameters requested.

1. Laboratory Data Audited:

Laboratory (Organization):

US EPA Region VIII Laboratory.

Sample Type(s)/Methods/Analyte(s): 524.2 Headspace (methane).

Sample Identification: PGDW5, PGDW20, PGDW30 and PGDW32.

WOs associated with these samples are identified in the support Excel Spreadsheets provided with this Audit of Data Quality Report.

QA Reviewers: David Gratson, Neptune and Company, Inc.

Method Information: Headspace (methane only analyte of interest): EPA Method 524.2, Region VIII OP ORGM-004 Methane by HS _DRAFT

File Information: Final Report included in file 8903002 final 16 jun 09.pdf.

Headspace (methane): Associated Files: 8903002 final 16 jun 09.pdf, 85622 - Headspace, 8270.pdf

QA/QC Criteria for Analytical Methods: QAPP specified and Laboratory specific QA/QC criteria and limits were used as the basis of this ADQ. Note the methane SOP was not finalized until after these data were analyzed.

Table 1. Region VIII Laboratory QA/QC Requirements for Headspace Methane.

QC Type	Headspace Methane	Frequency
Initial Calibration ICAL	Multi-level with $\leq 20\%$ RSD, or $r^2 \geq 0.99$. The lowest standard serves as the reporting limit.	Associated with each analytical batch, not required daily.
Method Blanks	Method blank prepared and compared against the reporting limit ($<RL$).	Each analytical batch, every 20 samples. Re-analyze all samples greater than 10 times the blank value if it is above the RL.
Initial Calibration Verification (ICV)	Verification of ICAL using a second source standard. % recovery within $\pm 30\%$ (70-130%).	Each analytical batch, recalibrate and rerun ICV
Continuing Calibration Verification (CCV)	A midpoint calibration standard analyzed every 10 sample and at the end of the analytical run. The %	Each analytical batch, every 10 samples. Re-analyze samples if criterion is not met.

	recovery within $\pm 20\%$ (80-120%).	
Laboratory Control Samples (LCS)	The % recovery within $\pm 30\%$ (70-130%).	Each analytical batch, every 20 samples. If recovery is low/high compare to MS and if available reprepare and analyze.
Matrix Spike/ MS Duplicate	Usually designated in the field. The % recovery within $\pm 30\%$ (70-130%).	Each analytical batch, every 20 samples. Reprepate and reanalyze MS if sample is available.
Storage Blanks	Same as for method blanks	Each set of field samples.
Reporting Limits	Lowest ICAL standard is at least as low as the reporting limit	Associated with each analytical batch.
Retention Time Windows	± 0.03 minutes	All samples within analytical batch that identify methane as analyte.

2. Summary of Assessment

OBSERVATIONS

1. **Matrix Spike and Matrix Spike Duplicates.** The headspace methane SOP (ORGM-04, Rev 0) Section 9.4 indicates that the batch QC is to include a matrix spike and matrix spike duplicate (MS/MSD), these are native samples spiked with methane. The selection is usually designated in the field. No MS/MSD combination was reported. All other QC indicated the analytical system was under quality control during the analyses of these samples.
2. **Storage Blank.** The headspace methane SOP (ORGM-04, Rev 0) Section 9.4 indicates that a storage blank is to be included in the sample set QC. Two blanks were analyzed in the analytical and preparation batch, see page 15 of 573 in the report. No identifier was located that indicated one of these blanks was a storage blank.

ITEMS REVIEWED – Headspace Methane Specific

Number	ADQ Issue	Yes	No	NA	Comments
File Information					
1	Provide File names: See Section 2.0 above.				
Sample Information					
2	Are samples uniquely identified and correctly transcribed throughout the data package to the summary of results?	X			Samples are uniquely labeled as PGDW5, PGDW20, PGDW30, and PGDW32 for all methods. In addition, samples are identified by unique Lab IDs throughout the final report and raw data packages for all methods.
3	Does sample collection documentation indicate that samples were collected as described in the QAPP, and the schedule and volumes in the planning documentation?	X Partia l			The only sample collection documentation within the report files is: date/time sample was collected and volume (for SVOC samples only).
4	Does sample collection documentation indicate appropriate preservation?	X			Samples were shipped on ice and cooler temperature monitors upon receipt read 4 °C.
5	If applicable, is chain-of-custody documentation complete? (Contains relinquished and received signatures, all samples identified with analyses, custody seals where appropriate)	X			COC documentation was found in files associated with specific work orders/batches. See Excel spreadsheet for methane for further details. Scanned images of the custody seals are included in the laboratory reports.

